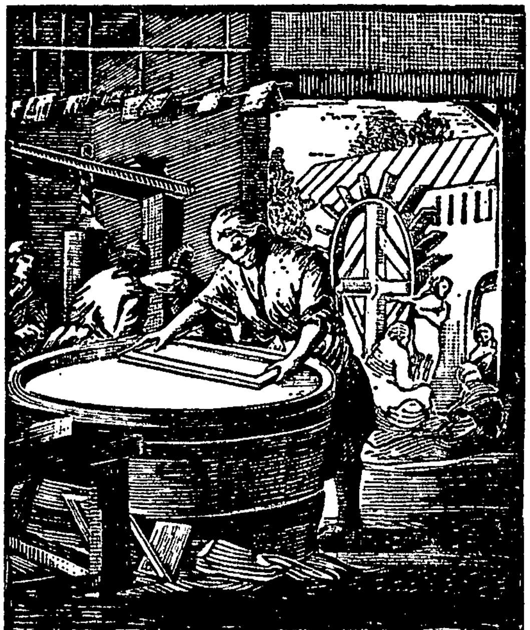

INSTITUTE OF PAPER CHEMISTRY
Appleton · Wisconsin

CHARACTERIZATION OF PULPS FOR PAPERMAKING

THE IPC COMPACTED FIBER DIMENSION APPARATUS

Project 2406

Report Two

A Progress Report

to

MEMBERS OF GROUP PROJECT 2406

October 18, 1965

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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TABLE OF CONTENTS

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CHARACTERIZATION OF PULPS FOR PAPERMAKING

THE IPC COMPACTED FIBER DIMENSION APPARATUS

SUMMARY

The Model II Compacted Fiber Dimension Apparatus (CFDA) is described. This instrument compacts a single pulp fiber between two optically flat anvils of synthetic sapphire with forces of 0-1000 grams (which can create stresses as high as 100,000 p.s.i. on short fiber segments). At a load high enough to collapse the fiber and create good optical contact between the sapphire compaction anvils and the surface of the fiber, the width and thickness of the resulting ribbonlike fiber are measured so that its compacted cross-sectional area may be computed. Width measurement is made, with a sensitivity of about 0.08 micron, by means of a Cooke (Dyson) image-splitting eyepiece. The thickness is measured, with a sensitivity of about 0.02 micron, by noting the separation of the sapphire pieces with the aid of an electrical transducer of the variable permeance type. It is shown that the calculated cross-sectional area of Douglas-fir latewood fiber segments decreases only slightly with increasing compacting stress after the stress has become great enough to collapse all visible voids within the fiber and between the fiber and compacting surfaces.

INTRODUCTION

This report describes an improved model of the IPC Compacted Fiber Dimension Apparatus (CFDA) for measuring the cross-sectional area (CSA) of compacted individual pulp fibers. The original apparatus (1, 2) was conceived and built when, as part of our pulp characterization program, it became desirable to have a rapid, accurate CSA measurement of each fiber tested on the IPC Fiber Load-Elongation Recorder (2, 3). This measurement would then permit the breaking stress of each individual fiber to be computed. The original CFDA was constructed to compress a single fiber between two flat, parallel glass plates with a force sufficient to collapse the fiber lumen and create good optical contact of the fiber with the glass plates across the whole width of the fiber. Measurement of the width of the resulting ribbonlike form was made by means of a special eyepiece on a microscope which viewed the fiber through the upper glass plate. An electrical transducer was used to determine the separation of the glass plates and, hence, the thickness of the compacted fiber. Then, by simple arithmetic, the CSA of the compacted fiber could be computed.

Although this first model worked reasonably well, it suffered from several deficiencies:

1. The glass plates were soft enough to be easily scratched on drawing the cleaning strip of glassine between them.

2. The glass had a low enough elastic modulus and the plates were thin enough to distort appreciably under the compaction load.

3. The fiber was viewed by reflected light, and there were times when insufficient light was available to permit seeing the flattened fiber clearly.

4. The mounting of the electrical transducer was not particularly rigid. This contributed to occasional erratic behavior of the thickness zero reading.

More disturbing, however, had been the behavior of the measured CSA as a function of the fiber segment length. As noted in Progress Report One, Project 2406 (2), the measured thickness of a given population of fibers decreased with decrease in segment length until a critical thickness was reached; then the measured thickness apparently increased with further decrease in segment length. As a result, the calculated CSA's followed the same general pattern. It was felt that this was not a real effect, but was probably caused by one or more of the apparatus shortcomings listed above. The most satisfactory way to work around this difficulty seemed to be to construct an improved model of the CFDA. This would allow not only correction of the problems listed above but also incorporation of a number of other design improvements. For these reasons, the Model II CFDA has been constructed.

DESCRIPTION OF THE APPARATUS

COMPACTION SYSTEM

Figure 1 is a photograph of the Model II CFDA; and Fig. 2, a closeup view of the compaction anvils (seen directly under the microscope objective lens). These anvils are made of clear, uncolored, synthetic sapphire, a material which possesses the desired hardness, stiffness, and optical clarity. The upper portion of the lower anvil – the "button" – is in the form of a truncated cone. Its optically-flat top surface, which is the area available for compaction, is 1/8 inch in diameter. The upper compaction anvil – the "plate" – has an optically flat lower surface which overlaps the top surface of the button on all sides. This configuration avoids the possibility of applying localized pressure at sharp edges of either piece, thus minimizing distortion and chipping. The plate is cantilevered above the button to provide unimpeded access to the compaction region.

The button is cemented with Eastman 910 adhesive into a shallow recess in the aluminum alloy baseplate, B, Fig. 1. The sapphire plate is cemented to the underside of the aluminum upper arm, C, with Eastman 910 cement and is reinforced by being bedded in a rigid epoxy resin in a stainless-steel yoke screwed to the underside of the upper arm.

The baseplate and upper arm are hinged together by means of a 1/16-inch-thick stainless-steel plate, D. The two threaded studs, E, permit the compacting surfaces of the sapphire anvils to be adjusted accurately parallel when in contact with each other. (Because of the simple hinge structure, there is a slight loss of parallelism when the anvils are separated. This is discussed later.)

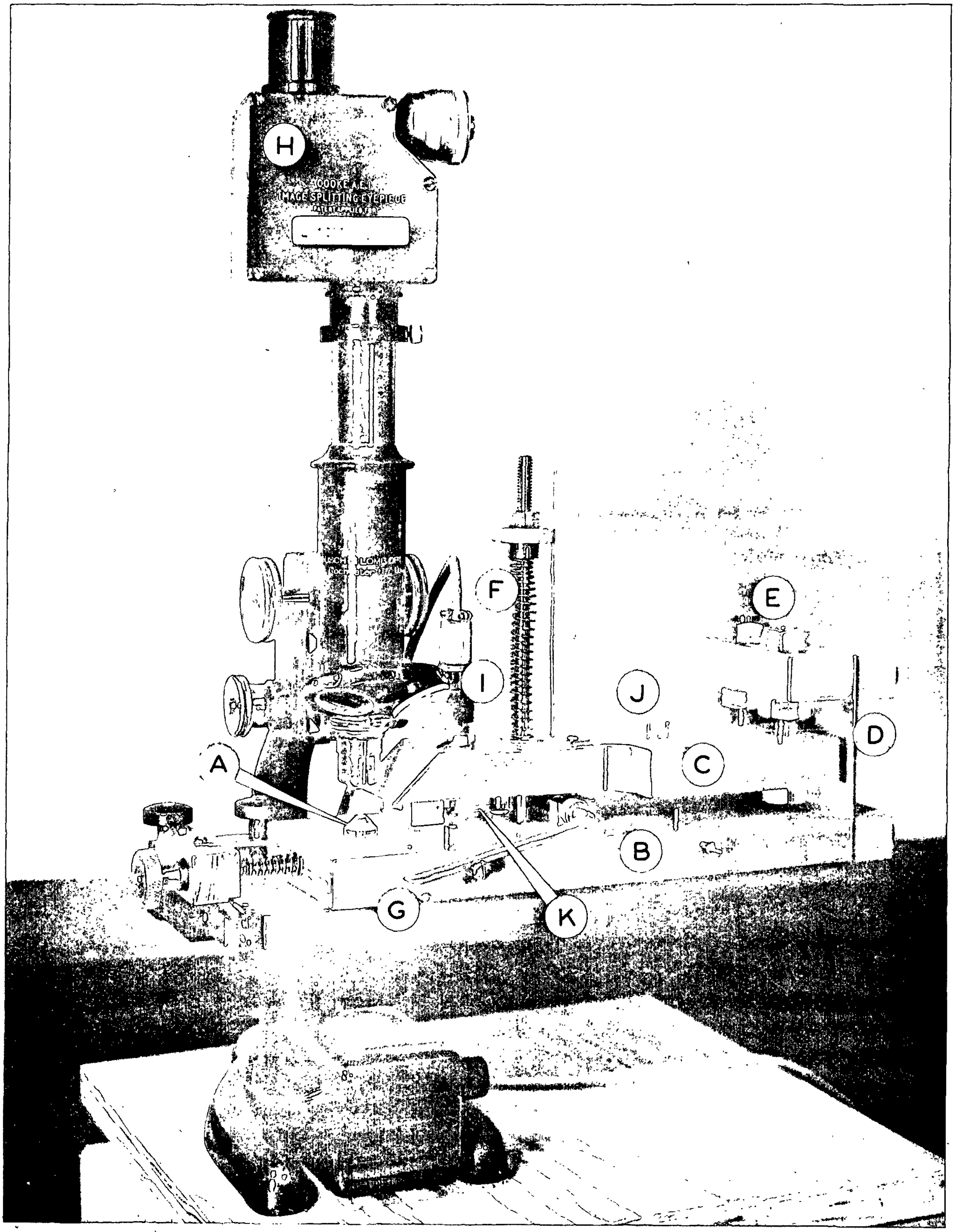

Figure 1. The IPC Compacted Fiber Dimension Apparatus (CFDA), Model II

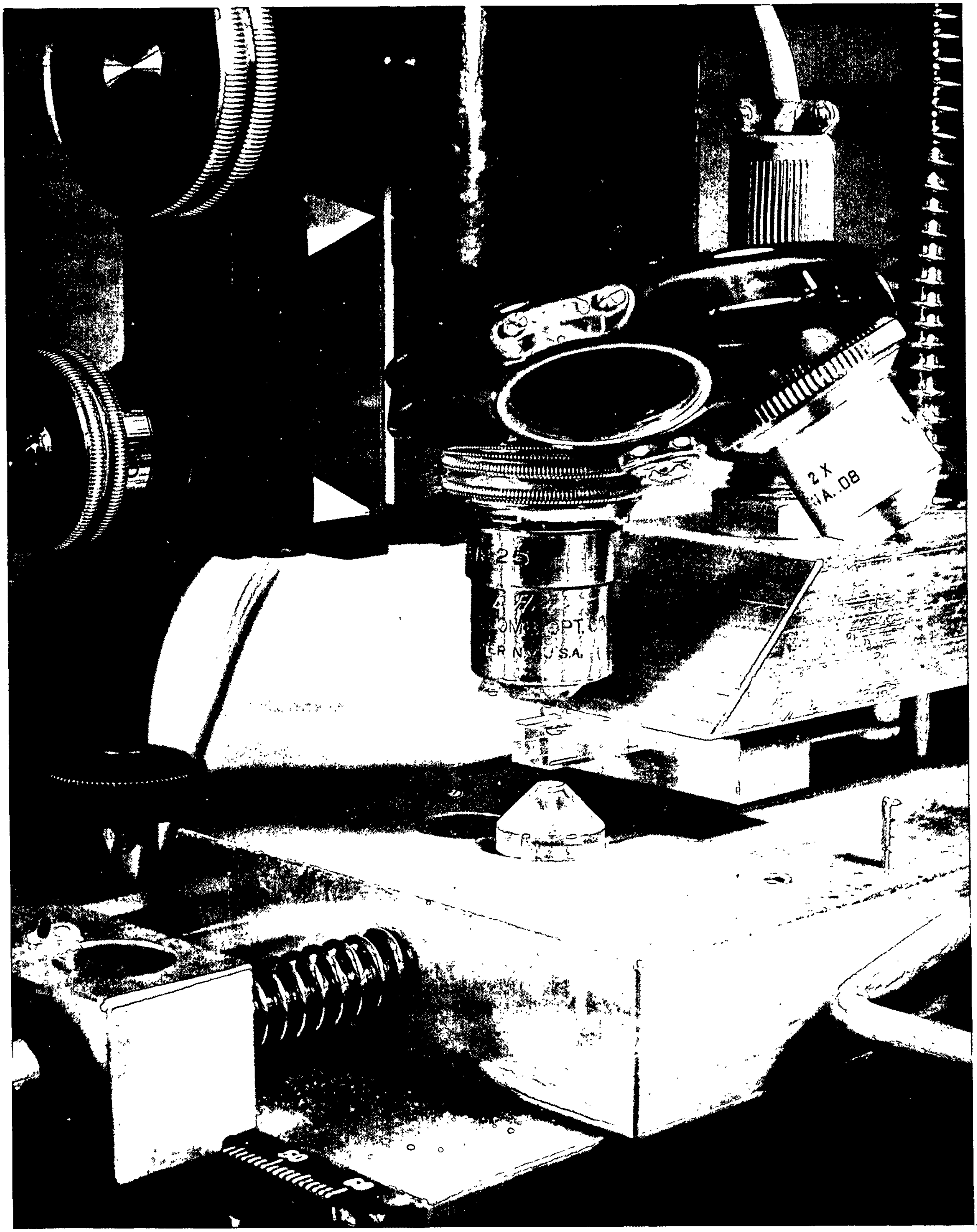

Figure 2. The Sapphire Compaction Anvils of the Model II CFDA
(Seen Directly Below the Microscope Objective Lens)

The compaction force is applied by means of the spring, F, which may be adjusted to various force levels by means of the thumbnut at its top end. A scale adjacent to the thumbnut is calibrated in units of force.

In order to conveniently separate the sapphire anvils to permit insertion and removal of the specimen, a rotary cam (operated by the handle, G) is located between the baseplate and the upper arm. Dimensions are such that, as seen in Fig. 2, the raised plate does not strike the microscope objective lens (10X) when focus is maintained on the top of the button.

WIDTH MEASUREMENT SYSTEM

The width of a fiber compacted between the sapphire anvils is measured with the aid of a Cooke (Dyson) image-splitting eyepiece, H, Fig. 1 (4). This device contains two prisms to produce two images of the object being viewed. A calibrated micrometer screw moves the prisms and thereby adjusts the relative separation of the images. A very accurate and precise measure of the width of the object being viewed is obtained by noting the difference between the micrometer reading when the images have been brought side by side to the position of just contacting each other and the micrometer reading when the images have been passed through each other until the opposite edges just touch.

Light for viewing originates in a substage microscope illuminator; it then passes through a hole in the special brass microscope stage, a hole in the baseplate, and through both sapphire anvils. Thus, the specimen is viewed by transmitted light intense enough to permit seeing the specimen clearly even when using the colored filters built into the Cooke eyepiece for providing greater contrast between the two images the eyepiece forms.

Positioning of the compacted fiber in the field of view of the microscope is accomplished with a mechanical stage modified to pivot and slide the relatively heavy compaction system on a pin projecting from the bottom of the baseplate near its hinged end. The pin rests in a groove in the microscope stage parallel to the long axis of the stage. A Teflon button under each corner of the opposite end of the baseplate rests on the stage, providing three-point support.

THICKNESS MEASUREMENT SYSTEM

The thickness of the compacted fiber is measured by sensing the separation of the sapphire anvils caused by the presence of the fiber. To accomplish this, the coil housing of a Crescent model KB-25 variable permeance transducer (5) is mounted in the upper arm (I, Fig. 1), and the transducer probe, which moves inside the coil housing, is positioned by the baseplate. The position of the probe relative to the housing is then detected by an auxiliary electrical system, shown schematically in Fig. 3. [This is the same system used with the Model I CFDA and is described in more detail in Reference (1).] Since there is only one position of the potentiometer slider which will balance the bridge for any given position of the transducer probe, the reading of a turns-counting dial on the balance potentiometer is a direct measure of the probe position. Hence, the difference in readings obtained when the compaction anvils are brought together with and without a fiber in place is a measure of the compacted fiber thickness.

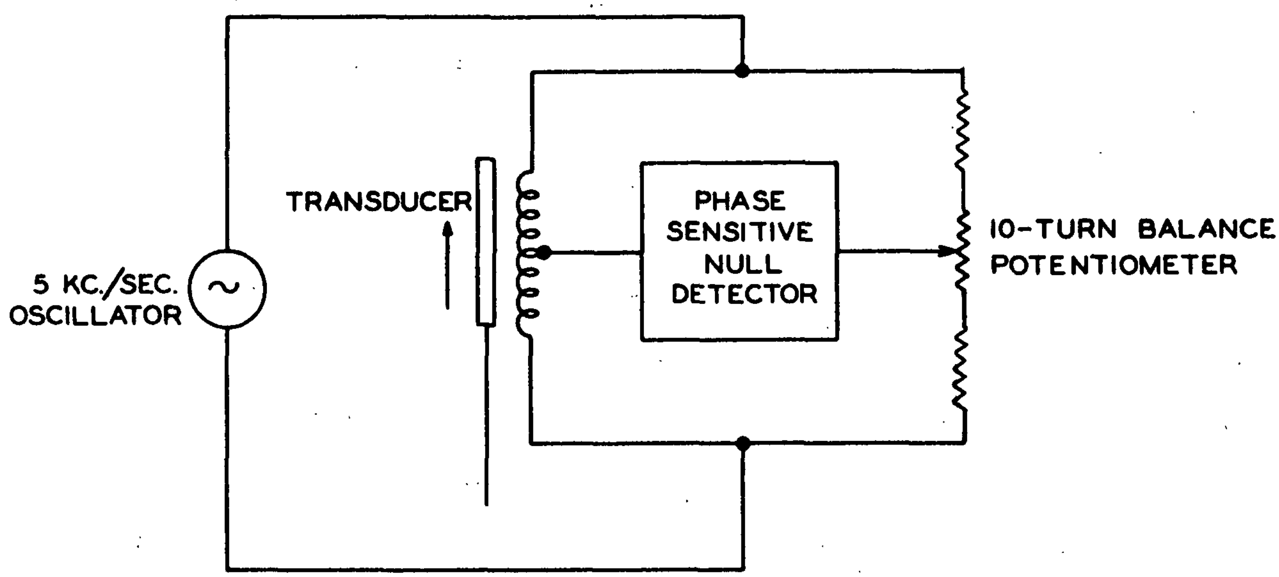

Figure 3. Elementary Schematic Diagram of the Electrical System for Measuring Thickness

ADJUSTMENT AND CALIBRATION

COMPACTION ANVILS

Parallelism and Distortion

After the CFDA had been in a 50% R.H., 73°F., room for a period long enough to establish thermal equilibrium, the compacting surfaces of the sapphire anvils were adjusted parallel by means of the two adjustment screws. Parallelism was observed by placing the compacting region in the field of view of a stereoscopic microscope, illuminating the region through one half of the microscope with the 546 nm. wavelength light from a mercury arc, and viewing the region through the other half of the microscope.

It was found possible to adjust the parallelism so that the interference pattern became one broad fringe and then disappeared completely as the anvils were brought into contact with each other. Further evidence of good parallelism is afforded by the observation that the clean anvil surfaces tend to "stick" together in much the same manner as do Johansson gage blocks. Changing the compaction force does not noticeably affect the parallelism, since an interference pattern did not reappear when the force was changed.

Because the anvils are simply hinged together, there is some loss of parallelism as they are separated. If a fiber of 10 microns compacted thickness (a fairly thick-walled fiber) is between the anvils, the separation at the edge of the 1/8-inch diameter compaction region farthest from the hinge will be about 0.15 micron greater than that at the edge nearest the hinge. This source of error is much smaller in actual use of the CFDA, however, because the fiber segment can be placed near the center of the compaction region or, if the fiber is long and extends through the central region, width measurement can be made in this central region. (The thickness calibration described later was done for the central region.)

With a single Douglas-fir latewood fiber between the sapphire anvils and compacted with a load of 1000 grams, one or two interference fringes were seen because of the hinge effect. These fringes, however, proceeded in straight lines across the whole compaction region and showed no evidence of local distortion of the sapphire anvils as they crossed the fiber.

Force

The flexure hinge between the upper arm and the baseplate is adjusted to keep the sapphire anvils slightly separated when the loading spring is not compressed. Then, with the loading spring compressed just enough to bring the pieces into contact, it is possible to apply light compacting loads by placing calibrated weights on a channel (J, Fig. 1) attached to the upper arm. Heavier loads are applied by removing the weights and further compressing the loading spring.

Calibration of the compaction force applied was made by supporting a trip beam balance above the CFDA, attaching a wire between the sapphire plate and one balance pan, applying a compaction force, and noting the weight added to the opposite balance pan when the thickness measuring system showed that the sapphire anvils had just begun to separate. Fiduciary marks have been made on the loading spring scale at loads of 0, 250, 500, 750, and 1000 grams; and a series of weights has been calibrated for placement on the upper arm for loads of less than 200 grams.

Cleaning

In order to obtain an accurate thickness measurement, it is necessary that the anvils be absolutely clean when brought together for the reading of "zero thickness." Many types of materials have been tried as cleaning wipers, including papers varying in hardness from lens tissue to glassine, Millipore filter papers, both synthetic and natural fiber cloths, plastic sheets, and chamois skin. In general, these materials work better dry than when wet with water or some other liquid. Air blasts, brushes, and elastomeric squeegees have also been tried, but no method has been found completely satisfactory.

The best cleaning seems to be done with a dry, well-used chamois skin, and this is the material presently being used. Half of the length of a strip of chamois, about 1-1/2 inches long, is placed between the sapphire anvils and the compaction load applied. The chamois is then carefully pulled in a direction parallel to the long axis of the CFDA and, before the end of the piece is reached, the cam is operated to remove the load. Pulling of the chamois is continuous until it has been pulled completely free of the sapphire anvils. In general, one pass is sufficient to remove any pulp fiber present and leave the compaction area free of debris, although occasionally the chamois will leave bits of its own fibers. These may be observed through the microscope and are removed by again drawing the chamois through.

THICKNESS MEASUREMENT

Stability

The electrical system for measuring the thickness of the compacted fiber is the same for the Model II CFDA as for the Model I (1), with the exception that the KB-25 transducer requires an oscillator frequency of 5 kc./sec. instead of

10 kc./sec. The stability of this system to changes of line voltage, oscillator frequency, and oscillator amplitude had been measured for the Model I, where it was found that, by use of a 0.1% line voltage regulator, drift due to these causes was at a negligibly low value.

Early in the testing of the Model II CFDA an attempt was made to measure the drift of the thickness indication after the clean sapphire anvils had been brought together with no specimen in place; this led to the discovery of large drifts in the indicated thickness "zero reading" whenever the anvils were separated and then brought together again. This behavior was eventually traced to the large excursion made by the transducer probe each time the anvils were separated. This excursion occurred because, in the interests of rigidity, the probe had been attached directly to the baseplate. The situation was corrected by mounting the probe on the end of a short, relatively stiff cantilever spring (K, Fig. 1) attached to the upper arm of the CFDA. With the anvils separated, the cantilever holds the probe very slightly below its readout position. An adjustable stop on the CFDA baseplate makes contact with and moves the probe only when the separation of the anvils is within the very small range for which thickness measurements are made. With this arrangement, probe excursion is sharply limited and no change in zero reading occurs when the anvils are separated and then brought together again. Also drift in zero reading over a 10-minute interval under steady load is no more than one balance dial division (0.017 micron, as shown in the next section).

Calibration

For calibration of the thickness measuring system, a 0.0001-inch dial micrometer was mounted above the compaction area by means of a rigid frame attached to the CFDA baseplate. The indicator rod of the micrometer moved vertically and rested on the upper surface of the sapphire plate directly over the center of the compaction area. The dial indicator itself had been proved previously to read accurately by attaching it to the frame of a Gaertner spectrum plate comparator and comparing the dial micrometer readings of carriage position with the comparator readings as the comparator carriage was moved.

Very thin shims of mica were used singly and in combination between the sapphire anvils to provide stable separation. At a given compacting load, readings of the null balance potentiometer and the dial micrometer (estimated to the nearest 0.00001 inch) were made as the mica shims were changed to cover the usable range. Plots of these data at load levels of 250, 500, 750, and 1000 grams were parallel straight lines, with a slope indicating a sensitivity of 0.01746 micron per null balance potentiometer dial division. The separation of the lines, however, indicates that the two readings of "zero" and "fiber" required for a fiber thickness measurement must be made at the same value of compacting force.

The accuracy of the thickness measurement is affected by the variability introduced by several factors:

	Null Balance Divisions
Long-term drift; effect eliminated by frequent zero readings	± 0
Stability of electrical system between successive readings	± 1
Fiber positioning; minimized by using central region of compaction area	± 2
Plate cleanliness (zero reading); minimized by careful cleaning	± 1

This gives a maximum error of four divisions, or about 0.07 micron.

WIDTH MEASUREMENT

Precision

The width of the compacted fiber is measured with the Cooke (Dyson) image-splitting eyepiece. Used with a 10X objective lens, the 15X magnification of the eyepiece itself gives an over-all optical magnification of about 150X.

To check the precision with which the Cooke eyepiece can be set, two runs were made with two different Douglas-fir earlywood fiber segments. For each run, one segment (about 0.4 mm. long) was compacted under a 500-gram load for one hour. Then the two settings for width measurement were made and recorded, these two settings being alternately made 30 times (a total of 120 settings for the two runs). The standard deviation of these readings indicates that the precision of measurement of the width of these fibers at any given point is extraordinarily good, the standard deviation being only 0.1 to 0.2% of the width.

Calibration

The micrometer of the Cooke eyepiece was calibrated by viewing a Bausch and Lomb stage micrometer scale (lines 0.001 inch apart etched on a glass slide) through the sapphire plate and taking micrometer readings of the distances between 1,2,...,10 lines. This gave a calibration factor of 0.784 micron per Cooke micrometer division. Thus, the sensitivity is approximately 0.08 micron when the micrometer setting is estimated to the nearest tenth of a division.

CSA EXPRESSION

When a fiber is compressed in the CFDA, its cross section may be assumed to appear as in Fig. 4. The CSA of such a shape is

$$A = (ab) - [b^2 - (\pi/4)b^2]$$

Using the calibration constants of the CFDA, this becomes

$$A = 0.001369TW - 0.0000654T^2$$

where

$\underline{A}$ = CSA, μ^2

$\underline{T}$ = thickness units from balance potentiometer dial

$\underline{W}$ = width units from Cooke micrometer.

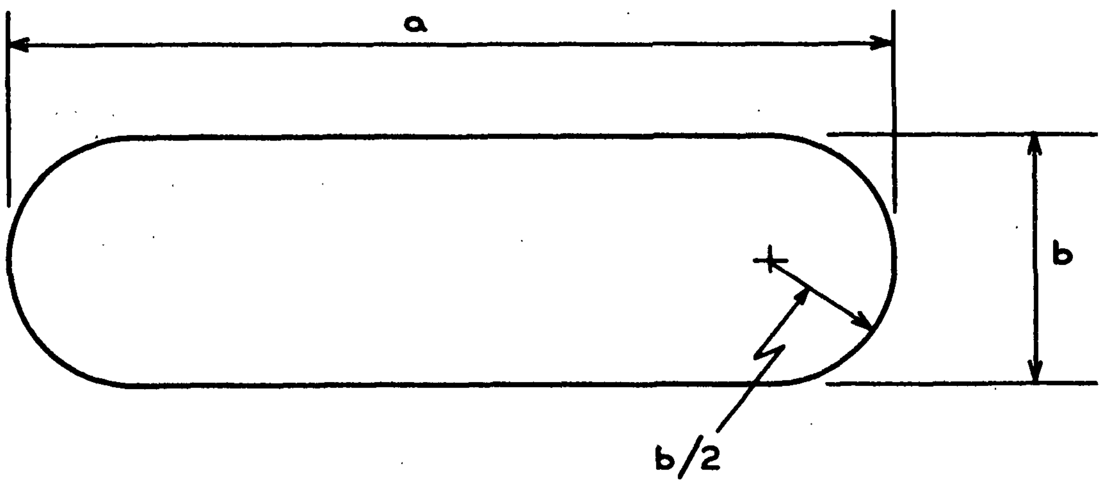

Figure 4. Assumed Shape of the Cross Section of a Compacted Fiber

MEASUREMENTS OF FIBERS

INSERTION TECHNIQUE

When a fiber load-elongation test has been conducted at a fairly long span (1 mm. or greater), it is possible to insert the longer of the broken fiber ends between the sapphire anvils while it is still glued to its mounting pin, then to bring the anvils together and pull on the pin to break it away from the fiber. However, this procedure runs the risk of chipping the sapphires if the metal pin should accidentally be included in the compaction area.

The preferred method of placing the broken fiber segment in the CFDA, whether it be a long or a short segment, is to cut it from the mounting pin using a very small, sharp knife. (A ground-down dissecting needle is satisfactory.) This cutting is done under a 15X stereoscopic microscope, allowing the cut end to fall onto a polished black glass plate. If the segment is long, it may be picked up with tweezers and deposited atop the sapphire button. If the segment is too short to be picked up with tweezers, it may be picked up with a wetted, single-hair brush for transfer to the sapphire button. In the latter case, before the anvils are brought together, several seconds are allowed for the excess water to evaporate and the fiber to come to equilibrium with the room atmosphere.

As a check of any possible effect on CSA of rewetting a fiber to transfer it to the CFDA, 47 of the longest Douglas-fir latewood fibers were cut approximately in half. One half was transferred dry to the CFDA and its CSA measured. The other half was then transferred wet to the CFDA and its CSA measured (after drying on the anvil). Figure 5 is a graph showing the per cent difference between the CSA's measured for the dry and wet transfer of the halves of each individual

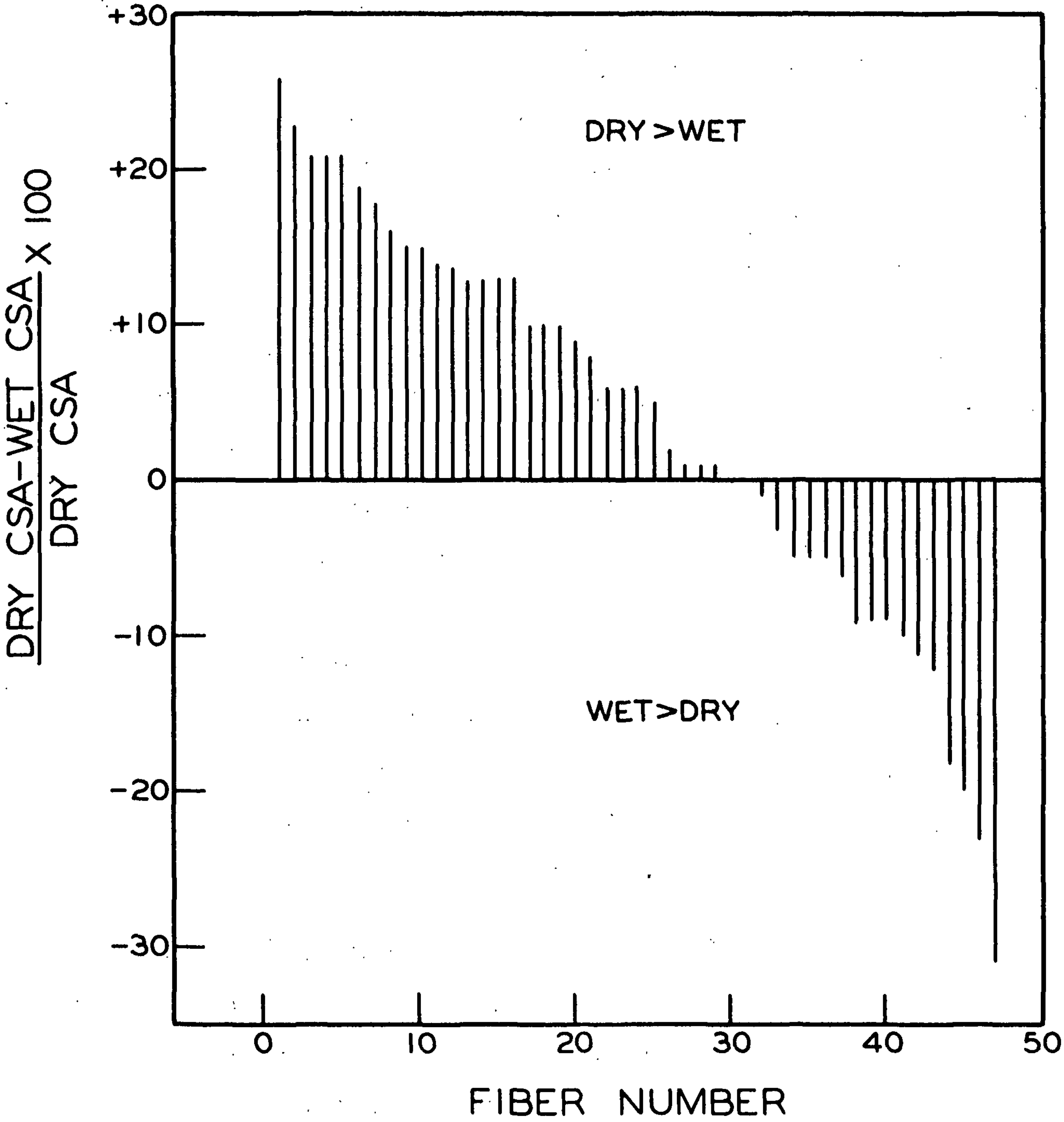

Figure 5. Difference in Cross-Sectional Area Between One Half of a Douglas-fir Latewood Fiber Transferred Dry to the CFDA and the Other Half of the Same Fiber Transferred Wet

fiber as related to that fiber's CSA after dry transfer. Although the average CSA of all the dry-transferred halves is about 5% greater than the average CSA of all the wet-transferred halves, the individual data show that either half can be greater. It seems probable, therefore, that the observed differences in CSA are due primarily to variations in the CSA of each individual fiber along its length, and that there is no significant difference between the CSA's measured after wet and dry transfer.

Since the broken fiber segments remaining after a load-elongation test at very short spans (0.3 mm. or less) are quite difficult to cut from the pins and place in the CFDA, a fairly long fiber to be tested at such a very short span is first cut in two. Half of it is then glued to the mounting pins for the load-elongation test while the other half is placed in the CFDA for CSA measurement.

COMPACTION FORCE VS. CSA

Several bundles of the long, kraft-pulped, chlorite-bleached, Douglas-fir, latewood fibers were embedded in collodion and microtomed to lengths of 0.2, 1, and 3 mm. with a sliding knife microtome. The collodion was then removed from the fiber segments by thorough washing with alcohol and ether.

One of the 3-mm. fiber segments (a length just long enough to extend completely across the compaction area) was then placed in the CFDA, the compaction load increased to 50 grams, and width and thickness measurements made. The load was then increased progressively to levels of 100, 250, 500, 750, and 1000 grams, with width and thickness measurements being made at each level. Following this, the fiber was removed and thickness zero readings made at each load level. This procedure was repeated until ten segments had been measured. Then ten fiber segments of 1 mm. and ten of 0.2-mm. length were measured in the same manner. In addition, measurement of the length of each 0.2-mm. segment was made at each load level.

The average CSA at each segment length and load level was then computed. Also, from the measurements of length and width, the area on which the compaction load acted was calculated so that the compaction stresses could be obtained. In Fig. 6, the average CSA of the fiber segments in each length group has been plotted against the average compacting stress for that group at each load level. The load levels are also noted on the figure. It is readily apparent that, as the compaction stress increases, the calculated CSA decreases, but tends to approach a constant value at the higher stresses. The trend of the points within any one segment length group is significant; but the absolute CSA level between groups differs because each group represents a slightly different population of fibers. (Also, the length-to-stress ratio at a given load varies somewhat between groups because of differences in average width.)

At the time the thickness and width measurements were made, observations of the degree of collapse of each fiber as a function of load were recorded. In general, the edges of the fiber were compacted sufficiently to be in good optical contact with the sapphire anvils at loads corresponding to compacting stresses of about 3 kg./mm.^{2}. At a stress of about 10 kg./mm.^{2}, the entire fiber, in general, was completely collapsed.

(Actually, the calculated stresses are correct only when the fiber is completely collapsed. At lesser degrees of collapse, the force is applied only to that smaller area which is in optical contact with the anvils; thus, the local stress may be much higher than that shown in Fig. 6.)

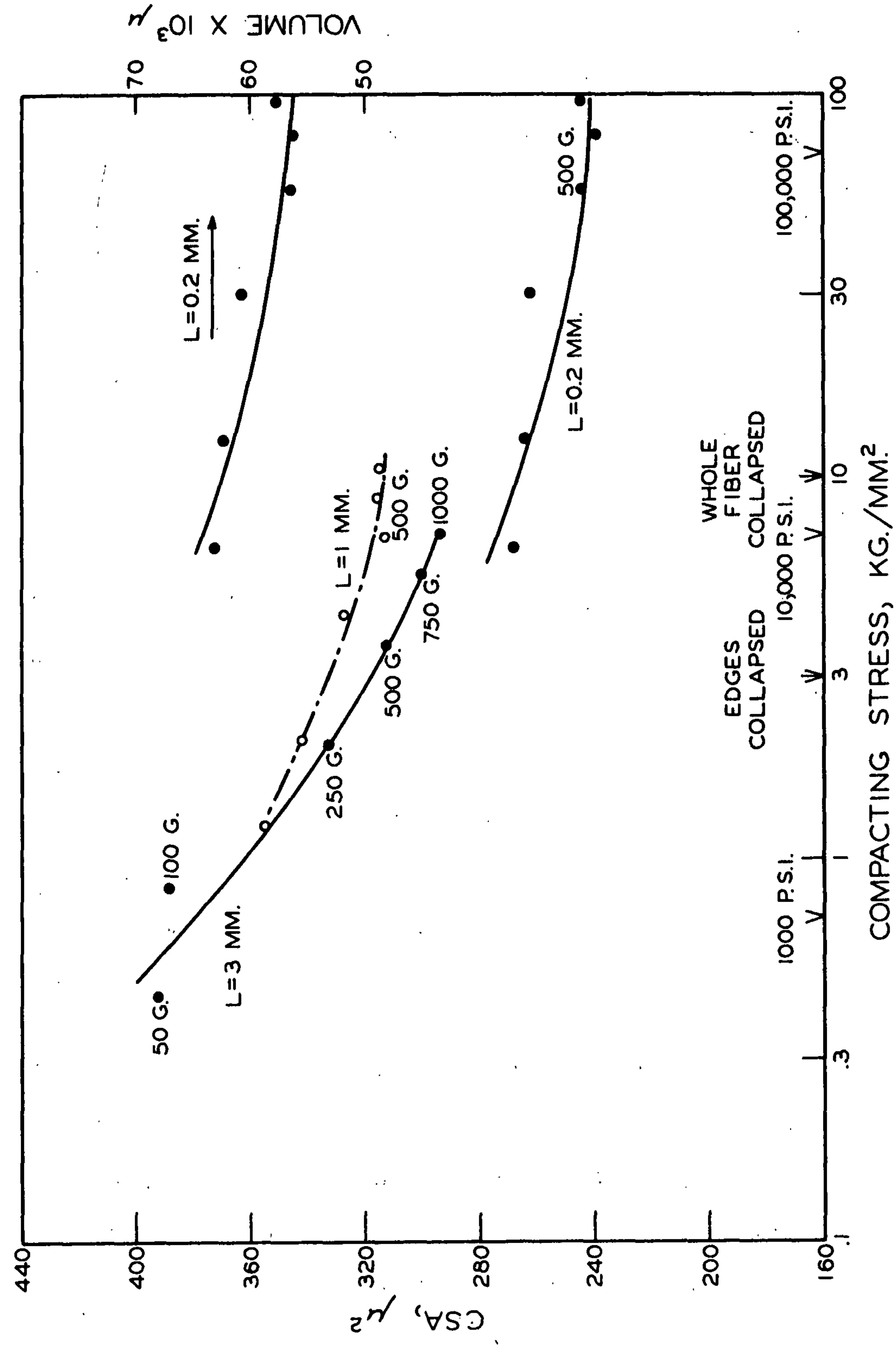

Figure 6. Cross-Sectional Area of Douglas-fir Latewood Fiber Segments vs. CFDA Compaction Stress. The Average of Ten Segments in Each of Three Segment Length Groups are Shown, as are the Compaction Loads. The Figure also Shows (a) the Stresses at Which the Edges of the Fibers and the Whole Fibers Were Collapsed and in Good Optical Contact with the Sapphire Compaction Anvils, and (b) a Plot of the Volume of the 0.2-mm. Segment Length Fibers vs. the Compaction Stress

It would appear from Fig. 6, then, that at stresses sufficient to completely collapse the fiber (or that region of the fiber at which width and thickness measurements are made), the measured CSA is probably very close to the ultimate compacted CSA. Since this stress is generally reached at a load level of 500 grams for the segment lengths commonly encountered, this load level has been chosen for routine use. (The normal compaction load of the Model I CFDA was 280 grams.)

With the short, 0.2-mm. segments, length measurements were made at each load level. This length was found to vary only slightly and in a random manner. Thus, the computed volumes of the fiber segments change with compacting pressure in direct relationship to changes in CSA. This is shown in Fig. 6.

SEGMENT LENGTH VS. CSA

Since the major difficulty with the Model I CFDA had been the apparent decrease and subsequent increase in CSA with decrease in the length of the fiber segment being measured, a test for this effect was made on the Model II. (This is, essentially, another aspect of the stress vs. CSA relationship.) The test was performed by cutting bundles of Douglas-fir earlywood fibers (embedded in collodion) to suitable lengths, removing the collodion by thorough washing with alcohol and ether, and measuring the thickness and width of the individual segments under a 500-gram compacting load. The results are plotted in Fig. 7.

Assuming that a fiber glued to pins for load-elongation testing would break near the middle of its span, half the span would represent the length of fiber segment available for CSA measurement. On this basis, the CSA vs. segment length relationship as found with the Model I CFDA during the study of the effect of span has been drawn in Fig. 7 for comparison.

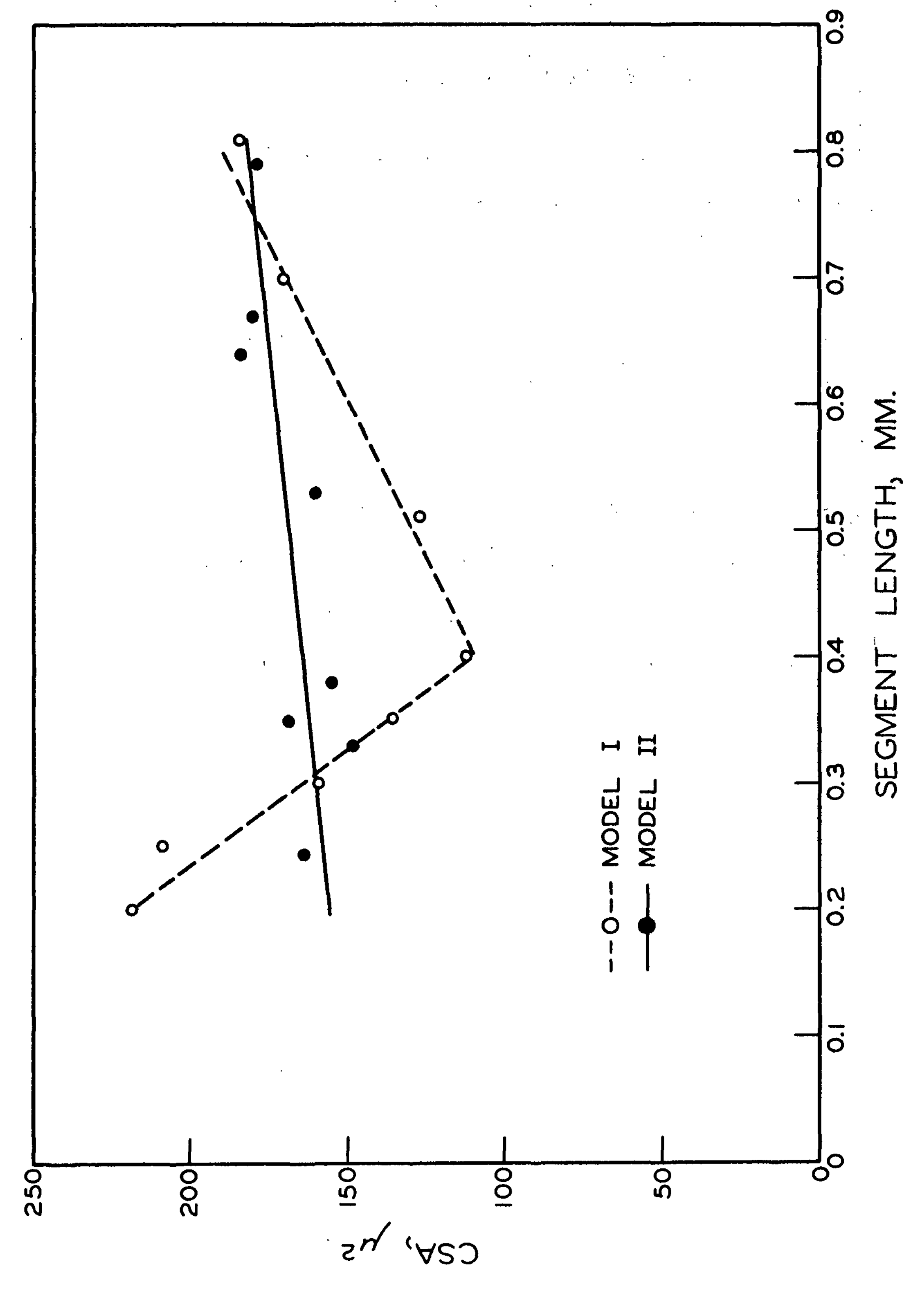

Figure 7. Cross-Sectional Area vs. Segment Length of Douglas-fir Earlywood Fibers as Measured with the Model I and the Model II CFDA

It would appear from this figure that the Model II CFDA shows a more logical relationship between CSA and segment length, and should, therefore, provide valid measurements of CSA at all segment lengths.

OTHER USES FOR THE APPARATUS

In addition to use of the CFDA for measuring the CSA of fibers *per se*, it has been suggested that loss of materials from fibers on extraction could be determined by noting the decrease in compacted CSA.

The apparatus also provides a tool to permit measurement of the lateral compressibility and conformability of wet and dry fibers, either singly or in crossed pairs.

Also, the improved model CFDA can be used for further study of the phenomenon (first observed with the Model I CFDA) of the expression of water from some fibers when compacted after initially being in equilibrium with relative humidities of 65% or higher.

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